

# § 60.647

# 40 CFR Ch. I (7–1–11 Edition)

rate shall be expressed in terms of equivalent sulfur mass flow rates (kg/hr (lb/hr)). The span of this monitoring system shall be set so that the equivalent emission limit of § 60.642(b) will be between 30 and 70 percent of the measurement range of the system. This requirement becomes effective upon promulgation of a performance specification for continuous monitoring systems for total reduced sulfur compounds at sulfur recovery plants.

(d) For those sources required to comply with paragraph (b) or (c) of this section, the average sulfur emission reduction efficiency achieved (R) shall be calculated for each 24-hour clock interval. The 24-hour interval may begin and end at any selected clock time, but must be consistent. The 24-hour average reduction efficiency (R) shall be computed based on the 24-hour average sulfur production rate (S) and sulfur emission rate (E), using the equation in § 60.644(c)(1).

(1) Data obtained from the sulfur production rate monitoring device specified in paragraph (a) of this section shall be used to determine S.

(2) Data obtained from the sulfur emission rate monitoring systems specified in paragraphs (b) or (c) of this section shall be used to calculate a 24-hour average for the sulfur emission rate (E). The monitoring system must provide at least one data point in each successive 15-minute interval. At least two data points must be used to calculate each 1-hour average. A minimum of 18 1-hour averages must be used to compute each 24-hour average.

(e) In lieu of complying with (b) or (c) of this section, those sources with a design capacity of less than 152 Mg/D (150 LT/D) of H<sub>2</sub>S expressed as sulfur may calculate the sulfur emission reduction efficiency achieved for each 24-hour period by:

$$R = \frac{K_2 S}{X}$$

Where:

R = The sulfur dioxide removal efficiency achieved during the 24-hour period, percent.

K<sub>2</sub> = Conversion factor, 0.02400 Mg/D per kg/hr (0.01071 LT/D per lb/hr).

S = The sulfur production rate during the 24-hour period, kg/hr (lb/hr).

X = The sulfur feed rate in the acid gas, Mg/D (LT/D).

(f) The monitoring devices required in paragraphs (b)(1), (b)(3) and (c) of this section shall be calibrated at least annually according to the manufacturer's specifications, as required by § 60.13(b).

(g) The continuous emission monitoring systems required in paragraphs (b)(1), (b)(3), and (c) of this section shall be subject to the emission monitoring requirements of § 60.13 of the General Provisions. For conducting the continuous emission monitoring system performance evaluation required by § 60.13(c), Performance Specification 2 shall apply, and Method 6 shall be used for systems required by paragraph (b) of this section.

[50 FR 40160, Oct. 1, 1985, as amended at 54 FR 6680, Feb. 14, 1989; 65 FR 61774, Oct. 17, 2000]

## § 60.647 Recordkeeping and reporting requirements.

(a) Records of the calculations and measurements required in § 60.642 (a) and (b) and § 60.646 (a) through (g) must be retained for at least 2 years following the date of the measurements by owners and operators subject to this subpart. This requirement is included under § 60.7(d) of the General Provisions.

(b) Each owner or operator shall submit a written report of excess emissions to the Administrator semiannually. For the purpose of these reports, excess emissions are defined as:

(1) Any 24-hour period (at consistent intervals) during which the average sulfur emission reduction efficiency (R) is less than the minimum required efficiency (Z).

(2) For any affected facility electing to comply with the provisions of § 60.646(b)(2), any 24-hour period during which the average temperature of the gases leaving the combustion zone of an incinerator is less than the appropriate operating temperature as determined during the most recent performance test in accordance with the provisions of § 60.646(b)(2). Each 24-hour period must consist of at least 96 temperature measurements equally spaced over the 24 hours.

(c) To certify that a facility is exempt from the control requirements of

these standards, each owner or operator of a facility with a design capacity less than 2 LT/D of  $H_2S$  in the acid gas (expressed as sulfur) shall keep, for the life of the facility, an analysis demonstrating that the facility's design capacity is less than 2 LT/D of  $H_2S$  expressed as sulfur.

(d) Each owner or operator who elects to comply with § 60.646(e) shall keep, for the life of the facility, a record demonstrating that the facility's design capacity is less than 150 LT/D of  $H_2S$  expressed as sulfur.

(e) The requirements of paragraph (b) of this section remain in force until and unless EPA, in delegating enforcement authority to a State under section 111(c) of the Act, approves reporting requirements or an alternative means of compliance surveillance adopted by such State. In that event, affected sources within the State will be relieved of obligation to comply with paragraph (b) of this section, provided that they comply with the requirements established by the State.

**§ 60.648 Optional procedure for measuring hydrogen sulfide in acid gas—Tutwiler Procedure.<sup>1</sup>**

(a) When an instantaneous sample is desired and  $H_2S$  concentration is ten grains per 1000 cubic foot or more, a 100 ml Tutwiler burette is used. For concentrations less than ten grains, a 500 ml Tutwiler burette and more dilute solutions are used. In principle, this method consists of titrating hydrogen sulfide in a gas sample directly with a standard solution of iodine.

(b) *Apparatus.* (See Figure 1.) A 100 or 500 ml capacity Tutwiler burette, with two-way glass stopcock at bottom and three-way stopcock at top which connect either with inlet tubulature or glass-stoppered cylinder, 10 ml capacity, graduated in 0.1 ml subdivision; rubber tubing connecting burette with leveling bottle.

(c) *Reagents.* (1) Iodine stock solution, 0.1N. Weight 12.7 g iodine, and 20 to 25 g cp potassium iodide for each liter of solution. Dissolve KI in as little water

as necessary; dissolve iodine in concentrated KI solution, make up to proper volume, and store in glass-stoppered brown glass bottle.

(2) Standard iodine solution, 1 ml=0.001771 g I. Transfer 33.7 ml of above 0.1N stock solution into a 250 ml volumetric flask; add water to mark and mix well. Then, for 100 ml sample of gas, 1 ml of standard iodine solution is equivalent to 100 grains  $H_2S$  per cubic feet of gas.

(3) Starch solution. Rub into a thin paste about one teaspoonful of wheat starch with a little water; pour into about a pint of boiling water; stir; let cool and decant off clear solution. Make fresh solution every few days.

(d) *Procedure.* Fill leveling bulb with starch solution. Raise (L), open cock (G), open (F) to (A), and close (F) when solutions starts to run out of gas inlet. Close (G). Purge gas sampling line and connect with (A). Lower (L) and open (F) and (G). When liquid level is several ml past the 100 ml mark, close (G) and (F), and disconnect sampling tube. Open (G) and bring starch solution to 100 ml mark by raising (L); then close (G). Open (F) momentarily, to bring gas in burette to atmospheric pressure, and close (F). Open (G), bring liquid level down to 10 ml mark by lowering (L). Close (G), clamp rubber tubing near (E) and disconnect it from burette. Rinse graduated cylinder with a standard iodine solution (0.00171 g I per ml); fill cylinder and record reading. Introduce successive small amounts of iodine thru (F); shake well after each addition; continue until a faint permanent blue color is obtained. Record reading; subtract from previous reading, and call difference D.

(e) With every fresh stock of starch solution perform a blank test as follows: introduce fresh starch solution into burette up to 100 ml mark. Close (F) and (G). Lower (L) and open (G). When liquid level reaches the 10 ml mark, close (G). With air in burette, titrate as during a test and up to same end point. Call ml of iodine used C. Then,

Grains  $H_2S$  per 100 cubic foot of gas=100 (D—C)

<sup>1</sup>Gas Engineers Handbook, Fuel Gas Engineering Practices, The Industrial Press, 93 Worth Street, New York, NY, 1966, First Edition, Second Printing, page 6/25 (Docket A-80-20-A, Entry II-1-67).